

(12) UK Patent Application (19) GB (11) 2 295 454 (13) A

(43) Date of A Publication 29.05.1996

(21) Application No 9523856.4

(22) Date of Filing 22.11.1995

(30) Priority Data

(31) 06291506 (32) 25.11.1994 (33) JP

(71) Applicant(s)

Hitachi, Ltd.

(Incorporated in Japan)

6 Kanda-Surugadai 4-chome, Chiyoda-ku, Tokyo,
Japan

Hitachi Instruments Engineering Co Ltd

(Incorporated in Japan)

832-2 Nagakubo, Horiguchi, Hitachinaka-shi,
Ibaraki-ken, Japan

(72) Inventor(s)

Shunsuke Koshihara

(51) INT CL⁶

H01J 37/244 , G01N 23/225 , G01T 1/36 , H01J 37/26

(52) UK CL (Edition O)

G1A AA6 ACK AR1 AR2 AT23 AT3
U1S S1910

(56) Documents Cited

GB 0966717 A US 5065020 A

(58) Field of Search

UK CL (Edition O) G1A ACDR ACK AHSR ATJ
INT CL⁶ G01N 23/225 , G01T 1/36 , H01J 37/244
37/252 37/26
On-Line: WPI

(72) cont

Mitsugu Sato
Naomasa Suzuki

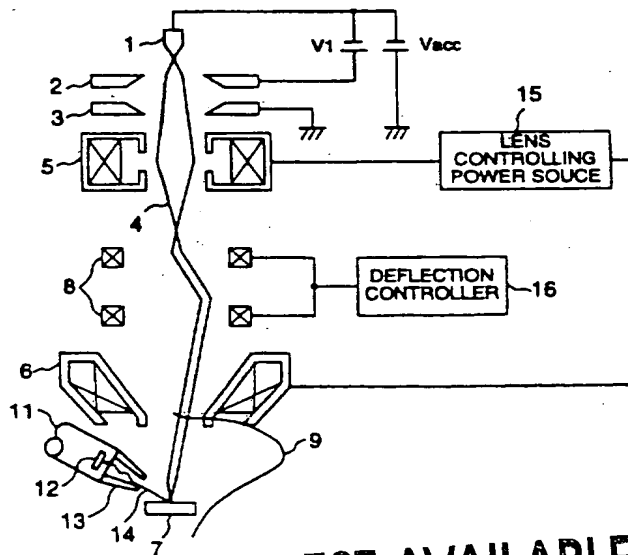
(74) Agent and/or Address for Service

Mewburn Ellis
York House, 23 Kingsway, LONDON, WC2B 6HP,
United Kingdom

(54) Energy dispersive x-ray analyzer

(57) In an electron microscope employing an X-ray spectrometer 11 according to the present invention, a collimator 13, made of non-magnetic material, is provided in the head portion of the spectrometer, and a part of the collimator is arranged in a leakage magnetic field 9 of an objective lens 6 included in the electron microscope, whereby the orbits of scattering electrons are curved and thus prevented from colliding with the X-ray spectrometer, thereby removing background noise from the X-ray spectrum. Modifications include the use of different materials and an irregularity structure on the inner surface of the collimator, and shaping the collimator so that its input aperture is narrower than its exit.

FIG.2



BEST AVAILABLE COPY

GB 2 295 454 A

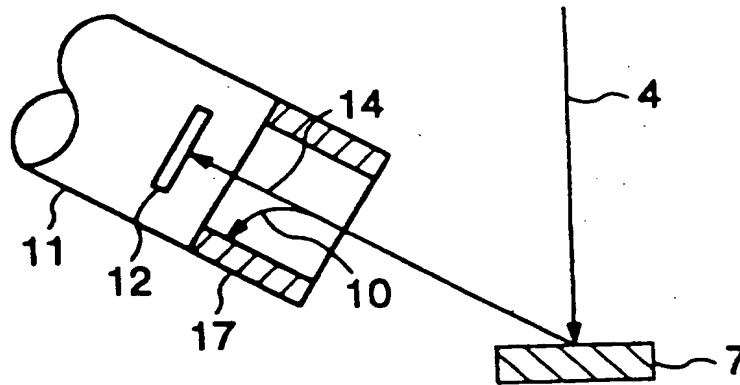


FIG.2

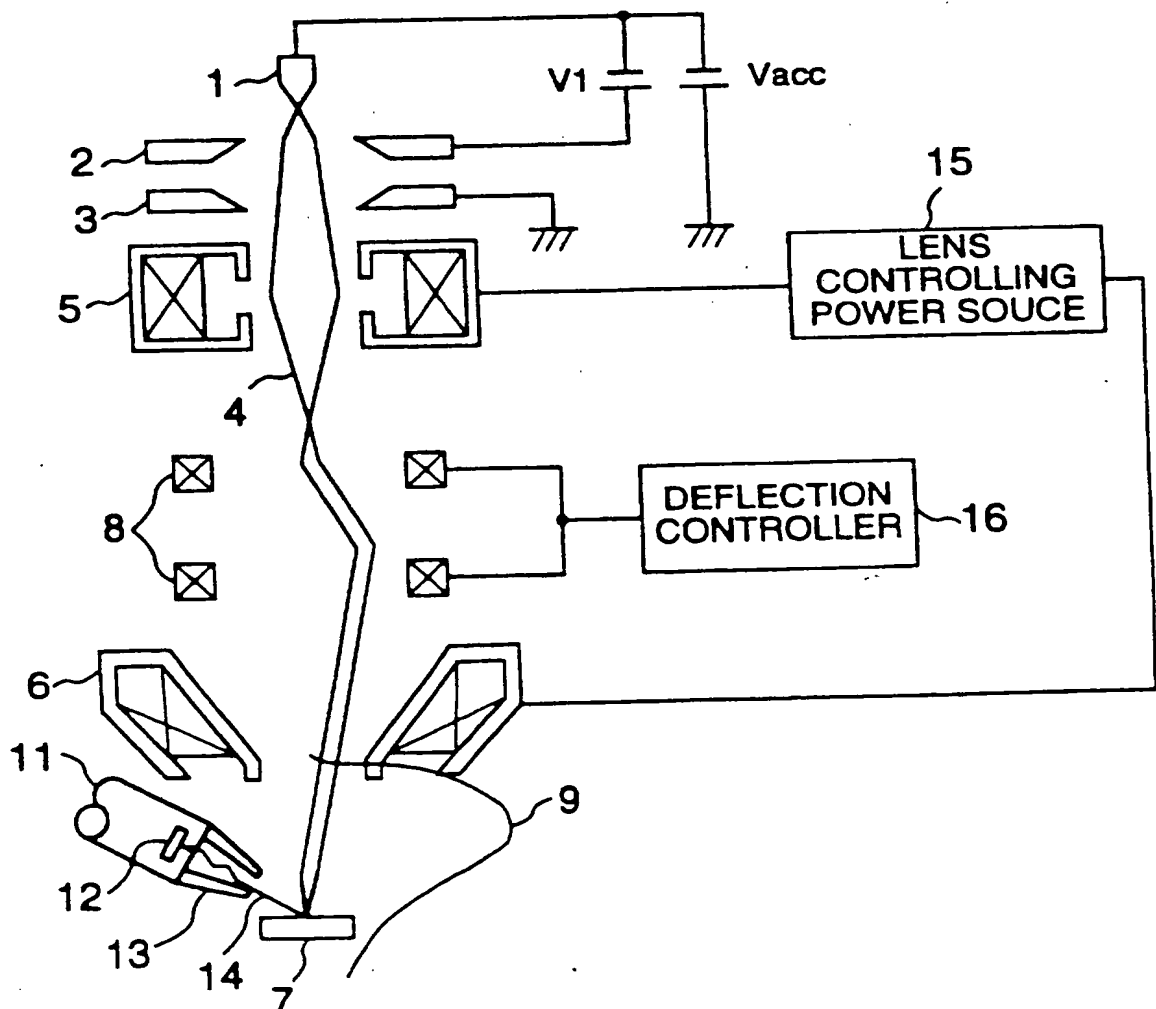


FIG.3

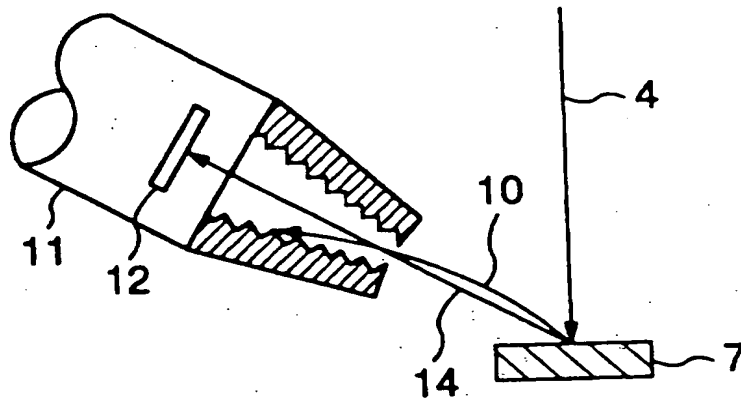
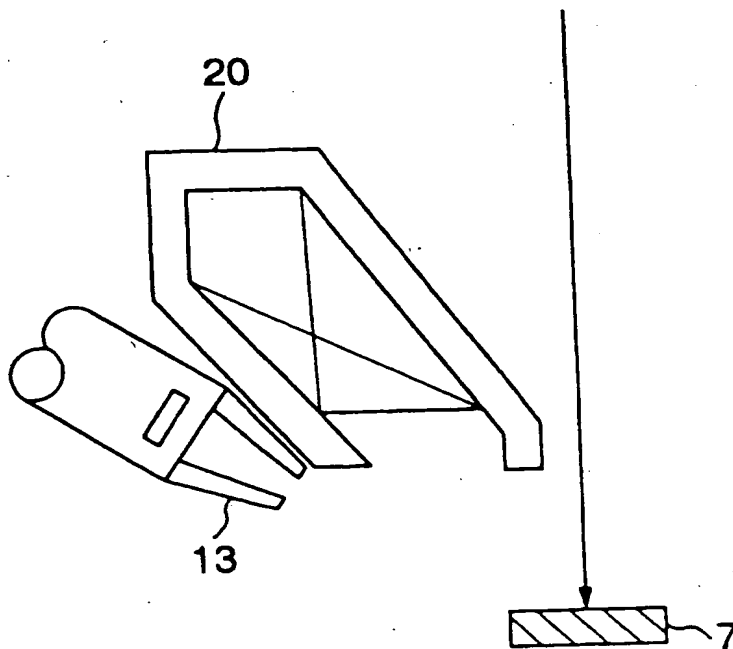


FIG.4



ENERGY DISPERSIVE X-RAY ANALYZER

The present invention relates in general to an energy dispersive X-ray analyzer, and more particularly to an energy dispersive X-ray analyzer which is capable
5 of detecting the X-rays at high sensitivity without influencing on the resolution of a system even when mounted to a high resolution scanning electron microscope having an objective lens of an inlens type or an objective lens of a magnetic field leakage type
10 attached thereto, and also which is suitable for performing the high precision X-ray analysis.

In order to perform the high precision X-ray analysis in an energy dispersive X-ray analyzer (hereinafter, referred to as "an EDX" for short, when
15 applicable) employing an energy dispersion X-ray spectrometer such as a silicon semiconductor detector, it is necessary to detect the X-rays which are emitted from a sample at high sensitivity and also to remove the scattering electrons (the reflected electrons) which are
20 emitted attendantly from the sample due to the collision of the incident electron beams with the sample. Those reflected electrons become the background noises in the X-ray spectrum and also influence on the precision or the like in the quantitative analysis.

As shown in Fig. 1, in a detector included in a conventional EDX, in order to remove the scattering electrons 10 which pass, together with the X-rays 14, into an EDX device 12 as an X-ray spectrometer from a sample 7, a ring-like permanent magnet 17 is provided as an electron trap in the head portion of the EDX device 12 and the orbits of the scattering electrons 10 are curved by the magnetic field which is generated by that permanent magnet, thereby preventing the scattering electrons from entering into the EDX device.

In order to perform the high resolution observation with respect to the sample, the scanning electron microscope of a system in which the sample is arranged in the magnetic field generated by the objective lens (the inlens system or the magnetic field leakage system) is often used. However, in such a scanning electron microscope, since the sample is arranged in the magnetic field of the objective lens, if the EDX spectrometer which has the permanent magnet as the electron trap in the head portion of the X-ray spectrometer is brought close to the vicinity of the sample, the magnetic field of the objective lens is disturbed by the magnetic field which is generated from the permanent magnet provided in the head portion of the X-ray spectrometer, and hence the high resolution observation can not be performed. This is a problem inherent in the prior art. As a result, in such a high resolution scanning electron microscope, the EDX

spectrometer can not be brought close to the sample, and hence the high sensitivity X-ray analysis and the high resolution observation can not be compatible with each other. This is another problem inherent in the prior art.

The present invention was made in order to mitigate the above-mentioned problems inherent in the prior art, and preferably it is an object of the present invention to provide an energy dispersive X-ray analyzer which is capable of performing the X-ray analysis at high sensitivity without harming the lens performance (the resolution) of the scanning electron microscope even when combined with the scanning electron microscope having the inlens type objective lens or the magnetic field leakage type objective lens.

In a first aspect, the present invention provides an energy dispersive X-ray analyzer including means for emitting electron beams, a condenser lens for condensing the electron beams, a magnetic field type objective lens, and an energy dispersive X-ray spectrometer, the X-rays which have been emitted from a sample arranged in a leakage magnetic field of said magnetic field type objective lens by irradiation of electrons being detected by said energy dispersive X-ray spectrometer, wherein a collimator made of a non-magnetic material is provided between said energy dispersive X-ray spectrometer and the sample, and at least part of said collimator is arranged in the leakage magnetic field of said magnetic field type objective lens.

In a second aspect, the present invention provides an energy dispersive X-ray analyzer including means for emitting electron beams, a condenser lens for
5 condensing the electron beams, a magnetic field type objective lens, and an energy dispersive X-ray spectrometer, the X-rays which have been emitted from a sample arranged in a leakage magnetic field of said
10 magnetic field type objective lens by irradiation of electrons being detected by said energy dispersive X-ray spectrometer, wherein a collimator which is made of a non-magnetic material and is sharpened toward the sample is provided between said energy dispersive X-ray
15 spectrometer and the sample.

In a third aspect, the present invention provides an energy dispersive X-ray analyzer including means for emitting electron beams, a condenser lens for
20 condensing the electron beams, a magnetic field type objective lens, and an energy dispersive X-ray spectrometer, the X-rays which have been emitted from a sample arranged in a leakage magnetic field of said magnetic field type objective lens by irradiation of
25 electrons being detected by said energy dispersive X-ray detector, wherein a collimator which is made of a non-magnetic material and an inner wall of which has an irregularity structure is provided between said energy dispersive X-ray spectrometer and the sample.

30 In a fourth aspect, the present invention provides

an energy dispersive X-ray analyzer including means for emitting electron beams, a condenser lens for
5 condensing the electron beams, a magnetic field type objective lens, and an energy dispersive X-ray spectrometer, the X-rays which have been emitted from a sample arranged in a leakage magnetic field of said magnetic field type objective lens by irradiation of
10 electrons being detected by said energy dispersive X-ray spectrometer, wherein a collimator which is made of a non-magnetic material and has an opening portion, which is smaller than an opening portion provided on the side of said energy dispersive X-ray spectrometer,
15 is provided between said energy dispersive X-ray spectrometer and the sample.

Preferably,

the present invention employs an EDX spectrometer in which a cylindrical collimator made of a non-magnetic material, such as aluminium, which does not influence on the magnetic field generated by an objective lens is mounted to the head portion of an X-ray spectrometer. In this connection, a part or the whole collimator is arranged in the magnetic field of the objective lens and the orbits of the scattering electrons are curved by the magnetic field of the objective lens, thereby preventing

the scattering electrons from entering into the X-ray detector.

Preferably, in addition, an irregularity structure, that is \square \square structure, is provided on an inner wall of an X-ray passing hole of the collimator, whereby even if the scattering electrons the orbits of which have been curved by the magnetic field of the objective lens collide with the inner wall of the X-ray passing hole of the collimator, the electrons which are generated due to the collision of the scattering electrons with the inner wall of the collimator is prevented from entering into the X-ray detection unit. The irregularity structure may be either formed by threading the inner surface of the collimator or formed by roughening the inner surface thereof. It is sufficient that a height of the irregularity is about 0.1 mm.

Preferably, in addition, in order to reduce the amount of electrons which have been generated when the scattering electrons collide with the inner wall of the X-ray passing hole of the collimator, a material (such as carbon) from which only the less amount of electrons are generated due to the collision is applied to the inner wall of the X-ray passing hole of the collimator, or the collimator itself is made of a material, such as aluminium, from which only the less amount of electrons are generated due to the collision.

Preferably, further, in order to prevent the scattering electrons from entering into the EDX device as much as

possible, the size of the inlet port for the X-rays of the collimator is made narrower than that of the outlet port, i.e., the side in which the EDX device is arranged.

5 By virtue of the above-mentioned arrangement, the scattering electrons which have been generated from the sample by the irradiation of the electron beams are curved with the orbits thereof by the magnetic field of the objective lens and hence are prevented from entering
10 into the EDX spectrometer. Even if the scattering electrons the orbits of which have been curved by the magnetic field of the objective lens collide with the inner wall of the X-ray passing hole of the collimator, since the inner wall of the X-ray passing hole of the
15 collimator is made of the material from which only the less amount of secondary electrons are generated due to the collision of the scattering electrons with the inner wall of the collimator, the amount of secondary
20 electrons which are generated from the X-ray passing hole due to the collision of the scattering electrons is reduced every repetition of the collision. In addition, since the inner wall of the X-ray passing hole of the collimator has the irregularity structure, even if the secondary electrons are generated by the collision of
25 the scattering electrons with the inner wall of the X-ray passing hole of the collimator, those secondary electrons do not enter into the X-ray detector because

they are interrupted by the irregularity structure of the inner wall of the X-ray passing hole.

On the other hand, since the X-rays which have been generated from the sample go straight on irrespective of the presence of the magnetic field of the objective lens, the X-rays thus generated are detected by the EDX spectrometer. Thus, according to the present invention, even when the X-ray spectrometer is mounted in the scanning electron microscope in which the sample is arranged in the magnetic field of the objective lens, the X-ray analysis can be performed at high sensitivity without influencing on the resolution of the scanning electron microscope.

Preferably, in addition, by making the size of the inlet port for the X-rays of the collimator narrow, it is possible that the scattering electrons hardly enter into the X-ray detector. By adopting this structure, since the large EDX device can be employed, the efficiency of detecting the X-rays can be further improved as compared with the case where the minor diameter of the cylindrical collimator is simply made narrow. Further, this structure has the shape which is suitable for arranging the collimator in the leakage magnetic field generated by the objective lens without being interrupted by the objective lens.

Preferably, furthermore, the collimator is made of a material having a small transmission factor for the X-rays, whereby it is possible to prevent the generation

of the secondary X-rays which will be excited and generated by applying the X-rays to the outer wall of the collimator. In addition, it is also possible to prevent the generation of the X-rays which will be
5 excited and generated due to the collision of the scattering electrons with the collimator. Since when the collimator is sharpened as in the present invention, the much amount of scattering electrons will collide with the outer wall of the collimator, the above-
10 mentioned structure can be said to be the effective structure.

Fig. 1 is a schematic view showing a structure of a conventional EDX spectrometer;

15 Fig. 2 is a schematic cross sectional view, partly in block diagram, showing an arrangement of an embodiment of the present invention;

Fig. 3 is a schematic cross sectional view showing a structure of a collimator portion; and

20 Fig. 4 is a schematic view showing an arrangement of a collimator of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Fig. 2 is a schematic cross sectional view, partly in block diagram, showing an arrangement of an
25 embodiment of the present invention. Incident electron beams 4 which have been emitted from a cathode 1 by

applying a voltage V_1 across the cathode 1 and a first anode 2 are accelerated by a voltage V_{acc} applied to a second anode 3 to move into the subsequent lens system. Those incident electron beams 4 are concentrated as a small spot on a surface of a sample 7 by the function of both a condenser lens 5 and an objective lens 6 which are controlled by a lens controlling power source 15 and then the sample is scanned in a two dimensional manner with the electron beams thus concentrated through the function of two stage deflecting coils 8. A scanning signal of the deflecting coils 8 is controlled by a deflection controller 16 in accordance with the observation magnification. A magnetic field 9 of the objective coil 6 is generated on the sample side and hence the sample 7 is arranged in the magnetic field of the objective lens.

An energy dispersive X-ray (EDX) detector 11 includes an EDX device 12 and a collimator 13 which is arranged in the front of the EDX device 12. The collimator 13 is a cylindrical structure which is made of aluminium, for example, as a non-magnetic material. In this connection, a screw with about 0.1 mm height is threaded in the inner surface of the collimator 13. The EDX spectrometer 11 is arranged in such a way that the collimator 13 thereof is located in the magnetic field 9 generated by the objective lens 6.

The X-rays 14 which have been emitted from the sample 14 pass through an X-ray passing hole of the

11

collimator 13 and then are detected by the EDX device 12 provided in the EDX spectrometer 11. On the other hand, scattering electrons 10 which have been emitted from the sample 7 are, as shown in Fig. 3, curved with the orbits thereof by the magnetic field 9 of the objective lens 6 and hence can not pass through the X-ray passing hole of the collimator to collide with the inner wall of the collimator 13. The scattering electrons which have collided with the inner wall of the collimator 13 serve to generate the secondary electrons from the collision surface of the inner wall of the collimator, but can not move towards the EDX device 12 since the inner wall of the collimator has the irregularity structure. In addition, since the surface of the inner wall is made of a material from which the secondary electrons are hardly generated due to the collision of the scattering electrons 10 with the inner wall of the collimator, the possibility that the scattering electrons 10 enter into the EDX device 12 becomes very small due to the mutually potentiating effect of that material and the above-mentioned irregularity structure.

In addition, by making the collimator 13 of a material having a small transmission factor for the X-rays such as tungsten or tantalum, it is possible to prevent the generation of the secondary X-rays which will be excited and generated by applying the X-rays to the outer wall of the collimator 13. In addition, it is also possible to prevent the generation of the X-rays

which will be excited and generated by the collision of the scattering electrons with the collimator 13. In particular, since when the collimator 13 is sharpened as in the present invention, the much amount of electrons
5 collide with the outer wall of the collimator, this structure can be said to be the effective structure.

Since the collimator 13 is sharpened towards the sample as shown in Fig. 4 in the present invention, the collimator 13 can be readily brought close up to the
10 leakage magnetic field of the objective lens 20 without being prevented by the objective lens 20. In addition, by sharpening the collimator 13, the inlet port for the X-rays is made narrower than the outlet port for the X-rays. In other words, it is possible to reduce the
15 entrance of the scattering electrons into the EDX device 12. This structural necessary condition is different in the following point from the style in which the minor diameter of the cylindrical collimator is simply narrowed to form a narrow cylinder.

20 That is, there is obtained an effect in which by sharpening the collimator 13, not only the inlet port of the collimator 13 can be narrowed, but also the large EDX device 12 can be employed. In general, the X-rays which have been obtained by applying the incident
25 electron beams 4 to the sample 7 are emitted radially with the irradiation point of the incident electron beams on the sample as the center. In other words, if the whole minor diameter of the collimator is simply

narrowed to intend to obtain the same inlet diameter as that of the collimator of the present invention, the detection efficiency is further reduced as compared with the present invention by the amount of X-rays which
5 collide with the inner wall of the collimator between the inlet port of the collimator and the EDX device.

As described above, by sharpening the collimator, the collimator can be readily arranged in the leakage magnetic field of the objective lens, and
10 also it is possible to reduce the entrance of the reflected electrons into the collimator.

Incidentally, in the case where both the efficiency of preventing the entrance of the scattering electrons into the collimator 13 and the efficiency of
15 detecting the X-rays are taken into consideration, if the inner wall of the collimator 13 is formed along the straight line connecting the irradiation point of the incident electron beams on the sample 7 and the end portion of the EDX device 12, it is possible to provide
20 the collimator 13 which is capable of making the detection efficiency of the EDX device 12 maximum and also of holding the entrance of the scattering electrons into the collimator 13 to a minimum while maintaining that detection efficiency as it is.

25 While the example has been described in which the EDX detector 11 is mounted to the scanning electron microscope to which the objective lens of the magnetic field leakage type is attached, it should be noted that

even when the EDX spectrometer 11 is mounted to the scanning electron microscope to which the objective lens of the inlens type is attached, the same effects can be obtained.

5 As set forth hereinabove, according to the present invention, since the X-rays can be detected efficiently by removing the scattering electrons which have been emitted from the sample without influencing on the magnetic field generated by the objective lens, in
10 the X-ray analysis in which the EDX spectrometer is combined with the high resolution scanning electron microscope having the sample arranged in the magnetic field of the objective lens, there is obtained the effect that it is possible to perform the X-ray analysis
15 at high sensitivity without injuring the resolution of the scanning electron microscope.

CLAIMS;

1. An energy dispersive X-ray analyzer including means for emitting electron beams, a condenser lens for condensing the electron beams, a magnetic field type objective lens, and an energy dispersive X-ray spectrometer, the X-rays which have been emitted from a sample arranged in a leakage magnetic field of said magnetic field type objective lens by irradiation of electrons, being detected by said energy dispersive X-ray spectrometer,

wherein a collimator made of a non-magnetic material is provided between said energy dispersive X-ray spectrometer and the sample, and

at least part of said collimator is arranged in the leakage magnetic field of said magnetic field type objective lens.

2. An energy dispersive X-ray analyzer according to claim 1, wherein an inner wall of said collimator has an irregularity structure.

3. An energy dispersive X-ray analyzer according to claim 1, wherein said collimator is sharpened towards the sample.

4. An energy dispersive X-ray analyzer according to claim 1, wherein said collimator is made of a material from which only the less amount of secondary electrons are emitted by the irradiation of the electrons.

5. An energy dispersive X-ray analyzer according to claim 1, wherein said collimator is made of a material having a small transmission factor for the X-rays.

6. An energy dispersive X-ray analyzer including means for emitting electron beams, a condenser lens for condensing the electron beams, a magnetic field type objective lens, and an energy dispersive X-ray spectrometer, the X-rays which have been emitted from a sample arranged in a leakage magnetic field of said magnetic field type objective lens by irradiation of electrons being detected by said energy dispersive X-ray spectrometer,

wherein a collimator which is made of a non-magnetic material and is sharpened towards the sample is provided between said energy dispersive X-ray spectrometer and the sample.

7. An energy dispersive X-ray analyzer according to claim 6, wherein at least part of said collimator is located in the leakage magnetic field of said magnetic field type objective lens.

8. An energy dispersive X-ray analyzer according to claim 6, wherein an inner wall of said collimator has an irregularity structure.

9. An energy dispersive X-ray analyzer according to claim 6, wherein said collimator is made of a material from which only the less amount of secondary

electrons are emitted by the irradiation of the electrons.

10. An energy dispersive X-ray analyzer according to claim 6, wherein said collimator is made of a material having a small transmission factor for the X-rays.

11. An energy dispersive X-ray analyzer including means for emitting electron beams, a condenser lens for condensing the electron beams, a magnetic field type objective lens, and an energy dispersive X-ray spectrometer, the X-rays which have been emitted from a sample arranged in a leakage magnetic field of said magnetic field type objective lens by irradiation of electrons being detected by said energy dispersive X-ray detector,

wherein a collimator which is made of a non-magnetic material and an inner wall of which has an irregularity structure is provided between said energy dispersive X-ray spectrometer and the sample.

12. An energy dispersive X-ray analyzer according to claim 11, wherein at least part of said collimator is located in the leakage magnetic field of said magnetic field type objective lens.

13. An energy dispersive X-ray analyzer according to claim 11, wherein said collimator is made of a material from which only the less amount of secondary electrons are emitted by the irradiation of the electrons.

14. An energy dispersive X-ray analyzer according to claim 11, wherein said collimator is sharpened towards the sample.
15. An energy dispersive X-ray analyzer according to claim 11, wherein said collimator is made of a material having a small transmission factor for the X-rays.
16. An energy dispersive X-ray analyzer including means for emitting electron beams, a condenser lens for condensing the electron beams, a magnetic field type objective lens, and an energy dispersive X-ray spectrometer, the X-rays which have been emitted from a sample arranged in a leakage magnetic field of said magnetic field type objective lens by irradiation of electrons being detected by said energy dispersive X-ray spectrometer,
- wherein a collimator which is made of a non-magnetic material and has an opening portion, which is smaller than an opening portion provided on the side of said energy dispersive X-ray spectrometer, is provided between said energy dispersive X-ray spectrometer and the sample.
17. An energy dispersive X-ray analyzer substantially as any one embodiment herein described with reference to Figures 2 to 4 of the accompanying drawings.

Patents Act 1977**Examiner's report to the Comptroller under Section 17
(The Search report)**19Application number
GB 9523856.4**Relevant Technical Fields**

(i) UK Cl (Ed.O) G1A (ACDR, ACK, AHSR, ATJ)

(ii) Int Cl (Ed.6) G01N (23/225); G01T (1/36);
H01J(37/244, 37/252, 37/26)**Databases (see below)**(i) UK Patent Office collections of GB, EP, WO and US
patent specifications.

(ii) ONLINE: WPI

Search Examiner
MR J STOKESDate of completion of Search
25 JANUARY 1996Documents considered relevant
following a search in respect of
Claims :-
1 TO 17**Categories of documents****X:** Document indicating lack of novelty or of
inventive step.**Y:** Document indicating lack of inventive step if
combined with one or more other documents of the
same category.**A:** Document indicating technological background
and/or state of the art.**P:** Document published on or after the declared priority
date but before the filing date of the present
application.**E:** Patent document published on or after, but with
priority date earlier than, the filing date of the present
application.**&:** Member of the same patent family; corresponding
document.

Category	Identity of document and relevant passages	Relevant to claim(s)
A	GB 0966717 (PHILIPS) page 1, lines 9-12, page 2, lines 50-53, Figure 1	
A	US 5065020 (HITACHI) Figure 1	

Databases: The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☒ **BLACK BORDERS**
- ☒ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☒ **FADED TEXT OR DRAWING**
- ☒ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☐ **SKEWED/SLANTED IMAGES**
- ☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☐ **GRAY SCALE DOCUMENTS**
- ☐ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.

This Page Blank (uspto)